

CALCIUM CARBONATE-FILLED ADDITION REACTION CURING TYPE SILICONE RUBBER COMPOSITION

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Abstract

PROBLEM TO BE SOLVED: To obtain a calcium carbonate-filled addition reaction curing type silicone rubber composition generating an extremely small amount of hydrogen during preservation.

SOLUTION: This calcium carbonate-filled addition reaction curing type silicone rubber composition contains (A) a diorganopolysiloxane containing at least two alkenyl groups in one molecule, (B) an organohydrogenpolysiloxane containing at least two hydrogen atoms binding with silicon atom in one molecule, (C) a platinum group metal-based catalyst and (D) a calcium carbonate powder surface-treated with a partially hydrolyzed condensed material of tetraalkoxysilane.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the hardened material obtained by hardening the addition-reaction hardening type silicone rubber constituent filled up with the calcium carbonate, its manufacture technique, and this constituent.

[0002]

[Description of the Prior Art] conventionally, a calcium carbonate is used for the purpose of raising properties, such as the thermal resistance of silicone rubber, and an electric insulation, -- having -- **** -- mainly -- an organic peroxidation -- the millable type silicone rubber by bridge formation, and condensation -- it is used for RTV silicone rubber by bridge formation however, an addition reaction -- it is not used for the silicone rubber by bridge formation The ground is that the alkali component is contained in the calcium-carbonate powder used for usual as a bulking agent as an impurity, and it reacts with the ORGANO hydro ***** polysiloxane by which this alkali component is contained as a cross linking agent in an addition-reaction hardening type silicone rubber constituent, and generates hydrogen gas. And when hydrogen gas is generated in this way, a constituent foams during a store or there is a risk of the internal pressure of the container which contained this constituent rising, and this container exploding.

[0003] without an addition-reaction hardening type silicone rubber constituent needs the long setting time like a condensed type RTV silicone rubber constituent on the other hand -- moreover, an addition reaction -- the silicone rubber by bridge formation -- condensation -- like RTV silicone rubber by bridge formation, since it is advantageous, the addition-reaction hardening type silicone rubber constituent filled up with the calcium carbonate is desired, in that [that the polysiloxane of a constituent cleaves in the elevated-temperature status under seal] the so-called ***** is not started However, the calcium-carbonate restoration addition-reaction hardening type silicone rubber constituent is not yet offered for the aforementioned ground.

[0004]

[Problem(s) to be Solved by the Invention] The purpose of this invention is to offer the hardened material obtained by hardening the addition-reaction hardening type silicone rubber constituent filled up with the calcium carbonate, its manufacture technique, and this constituent.

[0005]

[Means for Solving the Problem] this invention offers the calcium-carbonate restoration addition-reaction hardening type silicone rubber constituent containing the calcium-carbonate powder which carried out surface treatment by the diorganopolysiloxane which contains at least two alkenyl machines in (A) monad, the ORGANO hydro ***** polysiloxane which contains the hydrogen atom combined with (B) silicon atom in [at least two] a monad, (C) platinum group metal system catalyst, and the partial-hydrolysis condensate of (D) tetrapod alkoxysilane.

[0006] After this invention's being the manufacture technique of the aforementioned constituent and mixing calcium-carbonate powder and the partial-hydrolysis condensate of tetrapod alkoxysilane, heat-treat this mixture or Moreover, or part or all of the aforementioned (A) component, After mixing the partial-hydrolysis condensate of calcium-carbonate powder and tetrapod alkoxysilane, This mixture is heat-treated and the manufacture technique of the calcium-carbonate restoration addition-reaction hardening type silicone rubber constituent characterized by mixing the remaining component of a group which becomes this mixture from the aforementioned (A) component, (B) component, and (C) component is offered.

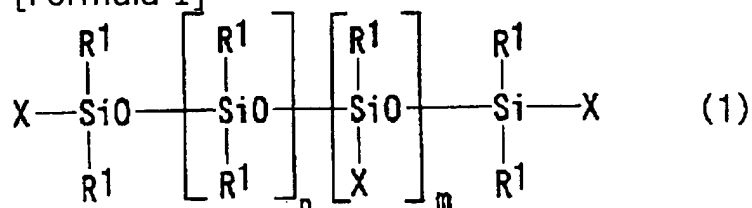
[0007] Moreover, this invention offers the hardened material obtained by hardening the aforementioned constituent.

[0008]

[Embodiments of the Invention] Hereafter, this invention is explained in detail.

[0009] (A) The alkenyl machine inclusion diorganopolysiloxane used for the constituent of an alkenyl machine inclusion diorganopolysiloxane this invention Although it is common that it is the thing of the shape of a straight chain by which chain both ends were blocked with the trio luganot siloxy machine by containing at least two alkenyl machines in a monad, and a principal chain fraction usually consisting of a repeat of a ***** siloxane unit fundamentally Although this may include the structure of the letter of branching in a part of molecular structure and you may be an annular solid, the diorganopolysiloxane of the shape of the point of physical properties, such as a mechanical strength of a hardened material, to a straight chain is desirable. Even if this alkenyl machine exists only in the both ends of a chain, it may exist in the middle of the both ends of a chain, and a chain. As an example of representation of such alkenyl machine inclusion diorganopolysiloxane, it is following general formula (1): [0010], for example.

[Formula 1]



The diorganopolysiloxane expressed with (R1 is the monovalent hydrocarbon radical of un-replacing [which does not contain an aliphatic unsaturated bond independently], or a substitute among a formula, X is an alkenyl machine, n is 0 or one or more integers, and m is 0 or one or more integers) is mentioned.

[0011] The inside of a formula, and R1 As a monovalent hydrocarbon radical of un-replacing [which does not contain an aliphatic unsaturated bond], or a

substitute For example, a methyl group, an ethyl group, a propyl group, an isopropyl machine, a butyl, An isobutyl machine, tert-butyl, a pentyl machine, a neopentyl machine, Alkyl group; cyclopentyl groups, such as a hexyl machine, a heptyl machine, an octyl machine, a nonyl machine, a decyl group, and the dodecyl, Cycloalkyl base; phenyl groups, such as a cyclohexyl machine and a cycloheptyl machine, Aryl group; benzyls, such as a tolyl group, a xylyl group, a naphthyl group, and a biphenylyl machine, A part or all of a hydrogen atom that has been combined with aralkyl machines [, such as a phenylethyl machine, a phenylpropyl machine, and a methyl benzyl,]; and the carbon atom of these bases A fluorine, The base replaced by halogen atoms, such as chlorine and a bromine, the cyano group, etc. For example, a methyl group, 2-ethyl group, 3-chloropropyl machine, 3, 3, and 3-trifluoropropyl machine, a chlorophenyl machine, a fluoro phenyl group, A cyano ethyl group, 3, 3, 4, 4, 5, 5, 6 and 6, and 6-nona fluoro hexyl machine etc. is mentioned. As for a typical thing, a carbon atomic number is [the carbon atomic number of 1-10, and especially a typical thing] the thing of 1-6. preferably A methyl group, an ethyl group, a propyl group, a methyl group, an ethyl group, It is the phenyl group of un-replacing, such as an alkyl group of un-replacing of the carbon atomic numbers 1-3, such as 3, 3, and 3-trifluoropropyl group and a cyano ethyl group, or a substitute and a phenyl group, a chlorophenyl machine, and a fluoro phenyl group, or a substitute.

[0012] Among a formula, as an alkenyl machine of X, for example, an about two to eight usual carbon atomic numbers [, such as a vinyl group an allyl group, a propenyl machine, an isopropenyl machine, a butenyl group, a hexenyl machine, and a cyclohexenyl group] thing is mentioned, and low-grade alkenyl machines, such as a vinyl group and an allyl group, are desirable especially.

[0013] n is 0 or one or more integers among a formula, and m is 0 or one or more integers. moreover, n and m are integers with which 10

$0 \leq n+m \leq 10,000$ are filled -- desirable -- more -- desirable -- 50

$0 \leq n+m \leq 2,000$ it is -- $0 \leq m/(n+m) \leq 0.2$ [and] It is the integer to satisfy.

[0014] Moreover, 10 - 1,000,000 cSt and the thing which is 100 - 500,000 cSt grade have [such alkenyl machine inclusion diorganopolysiloxane] the especially desirable viscosity in 25 degrees C.

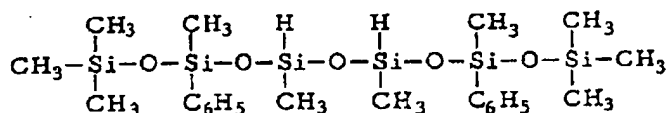
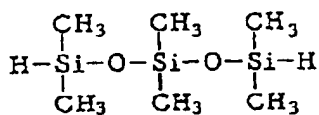
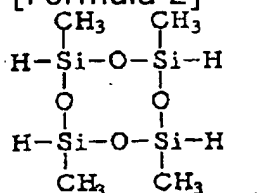
[0015] (B) The ORGANO hydro polysiloxane used for the constituent of the ORGANO hydro polysiloxane this invention may contain preferably the hydrogen atom (namely, SiH base) combined with three or more silicon atoms in [at least two] a monad, and any of the resin-like object of the shape of a straight chain, the letter of branching, annular, or a three-dimensions network structure are sufficient as it. As an example of representation of such an ORGANO hydro polysiloxane, it is the following average empirical formula (2), for example. : $\text{Ha R}_2 \text{b SiO} (4-a-b)/2$ (2)

The ORGANO hydro polysiloxane expressed with (R2 being the monovalent hydrocarbon radical of un-replacing [which does not contain an aliphatic unsaturated bond independently], or a substitute among a formula, and a and b being the numbers used as $0 < a < 2$, $0.8 \leq b \leq 2$, and $0.8 < a+b \leq 3$, and being a number preferably set to $0.05 \leq a \leq 1$, $1.5 \leq b \leq 2$, and $1.8 \leq a+b \leq 2.7$) is mentioned.

[0016] the inside of a formula, and R2 the monovalent hydrocarbon radical of un-replacing [which does not contain an aliphatic unsaturated bond], or a

substitute ***** -- R₁ of the aforementioned general formula (1) ***** -- what was illustrated, and the same thing mention -- having -- a typical thing -- a carbon atomic number -- 1-10 -- especially a carbon atomic number is the thing of 1-7, and is low-grade alkyl group [of the carbon atomic numbers 1-3, such as a methyl group,], phenyl-group, 3, 3, As an example of such an ORGANO hydro ***** polysiloxane For example, 1, 1, 3, and 3-tetramethyl disiloxane, 1, 3 and 5, a 7-tetramethyl tetracyclo siloxane, Siloxane oligomer; chain both-ends trimethylsiloxy machine blockade methyl hydrogen polysiloxane, such as 1, 3, 5, 7, and 8-pen reservoir chill pentacyclosiloxane, A chain both-ends trimethylsiloxy base blockade dimethyl siloxane methyl hydro ***** siloxane copolymer, Chain both-ends silanol-group blockade methyl hydrogen polysiloxane, A chain both-ends silanol-group blockade dimethyl siloxane methyl hydro ***** siloxane copolymer, Chain both-ends dimethyl hydrogen siloxy machine blockade dimethylpolysiloxane, Chain both-ends dimethyl hydrogen siloxy machine blockade methyl hydrogen polysiloxane, chain both ends --; R₂, such as a dimethyl hydrogen siloxy machine blockade dimethyl siloxane methyl hydro ***** siloxane copolymer, (H) SiO 1/2 A unit and SiO 4/2 It consists of a unit. It is R₃ SiO 1/2 arbitrarily. A unit and R₂ SiO 2/2 A unit and R(H) SiO 2/2 Unit, (H) SiO 3/2 A unit or RSiO 3/2 The silicone resin (however, the inside of a formula and R are the same as that of the monovalent hydrocarbon radical of un-replacing [which was illustrated as R₁ of the above] or a substitute) which may include a unit is mentioned, and it is following formula: [0017] further.

[Formula 2]



What is expressed with a grade is mentioned.

[0018] The ORGANO hydro ***** polysiloxane used for the constituent of this invention It can obtain by well-known technique, for example, they are following general formula: R₂ SiHCl₂ and R₂ 2 SiHCl (among a formula). R₂ The cohydrolysis of at least one sort of chlorosilicanes chosen out of being the same as that of the above is carried out. Or combining at least one sort of chlorosilicanes chosen out of this chlorosilicane, following general formula: R₂ 3 SiCl, and R₂ 2 SiCl₂ (the inside of a formula and R₂ are the same as that of the above), a cohydrolysis can be carried out and it can obtain. Moreover, what carried out the equilibration of the polysiloxane which carries out a cohydrolysis in this way and was obtained is sufficient as the ORGANO hydro ***** polysiloxane.

[0019] the amount of the component (B) used -- the alkenyl machine in the alkenyl machine inclusion diorganopolysiloxane of a component (A) -- an amount from which the hydrogen atom (namely, SiH base) combined with the silicon atom per mol and in the ORGANO hydro ***** polysiloxane of a component (B) usually becomes 0.5-4 mols -- desirable -- 1-2.5 It is the amount which serves as a mol.

[0020] (C) The platinum group metal system catalyst used for a platinum group metal system catalyst this invention is a catalyst for promoting the addition reaction with the hydrogen atom combined with the alkenyl machine of the aforementioned component (A), and the silicon atom of a component (B), and a well-known catalyst is mentioned as a catalyst used for a hydrosilylation reaction. As the example, for example Platinum group metal simple substance; H_2 PtCl₄ and nH_2 O, such as platinum (platinum black is included), a rhodium, and palladium, H_2 PtCl₆ and nH_2 O, NaHPtCl₆ and nH_2 O, KHPtCl₆ and nH_2 O, Na₂ PtCl₆ and nH_2 O, K₂ PtCl₄ and nH_2 O, PtCl₄ and nH_2 O, and PtCl₂, Na₂ HPtCl₄ and nH_2 O (however, n is the integer of 0-6 among a formula) the complex (the U.S. patent specification of No. 3,159,601 --) of a platinum chloride, such as being 0 or 6 preferably, a chloroplatinic acid and a chloroplatinate; alcoholic denaturation chloroplatinic-acid (refer to the U.S. patent specification of No. 3,220,972); chloroplatinic acid, and an olefin this No. 3,159,662 specification -- said -- the 3,775,452nd -- No. specification reference; platinum black -- The thing; rhodium-olefin complex; ***** tris (triphenylphosphine) rhodium (***** catalyst); platinum chloride which made support, such as an alumina, a silica, and carbon, support platinum group metals, such as palladium The complex with a chloroplatinic acid or a chloroplatinate and a vinyl-group inclusion siloxane, especially a vinyl-group inclusion cyclosiloxane etc. is mentioned.

[0021] In the so-called amount of catalysts, the amount of the component (C) used is good, is usually the weight conversion of a platinum group metal to the total quantity of a component (A) and a component (B), and is good at 0.1-500 ppm, especially about 0.5-200 ppm of **s.

[0022] (D) Carry out surface treatment of the calcium-carbonate powder used for the calcium-carbonate powder this invention which carried out surface treatment by the partial-hydrolysis condensate of tetrapod alkoxysilane. In the addition-reaction hardening type silicone rubber constituent containing the calcium-carbonate powder which performed such surface treatment, the dehydrogenation of the ORGANO hydro ***** polysiloxane resulting from an alkali component being eluted from this calcium-carbonate powder is reduced. Therefore, a ** (D) component prevents the foaming under store of this constituent, and is a thing in which the internal pressure of the container which contained this constituent rises, and this container explodes and which gives properties, such as thermal resistance and an electric insulation, to this constituent while carrying out thing prevention.

[0023] ** The tetrapod alkoxysilane used for the partial-hydrolysis condensate this invention of tetrapod alkoxysilane is expressed with following general formula: $Si(OR)_3R_4$ (the inside of a formula and R₃ are the alkyl groups of un-replacing or a low-grade alkoxy substitute independently). the inside of a formula, and R₃ the alkyl group of un-replacing or a low-grade alkoxy substitute ***** -- a methyl group, an ethyl group, a propyl group, an isopropyl machine, a butyl, an isobutyl machine, tert-butyl, a pentyl machine, a

neopentyl machine, a hexyl machine, a heptyl machine, an octyl machine, a nonyl machine, a decyl group, the dodecyl and a methoxymethyl machine, a methoxy ethyl group, an ethoxy methyl group, an ethoxy ethyl group, etc. -- usually -- carbon numbers 1-12 --

[0024] As a partial-hydrolysis condensate of such tetrapod alkoxysilane For example, following average empirical-formula: $[(OR_3)_3SiO_{1/2}]^K [SiO_{4/2}]^N [(OR_3)_2SiO_{2/2}]^L [(OR_3)SiO_{3/2}]^M$ (among a formula) R_3 the above -- the same -- K, L, M, and N -- $K+L+M+N=1$, $0.002 \leq K \leq 1$, $0 \leq L \leq 0.998$, and $0 \leq M \leq 0.998$ What is expressed is mentioned. General-formula [Following] (3): and the number with which it is satisfied of $0 \leq N \leq 0.35$ -- it is -- especially a typical thing $R_3 O-[Si(OR_3)_2 O]^j - R_3$ (3)

It is expressed with (R_3 is the same as that of the above among the formula, and j is one or more integers). For such a partial-hydrolysis condensate of tetrapod alkoxysilane, weight average molecular weight is 150-100,000. A thing is desirable.

[0025] ** Although a well-known thing can be used for the calcium-carbonate powder with which calcium-carbonate powder surface treatment is presented, its precipitated calcium carbonate preferably manufactured by the chemical technique is desirable at the point that purity is high. The mean particle diameter of calcium-carbonate powder is usually 0.1-30 micrometers. It is easy to be the thing of a grade and can choose suitably by intended use.

[0026] ** As the technique of surface treatment surface treatment, a well-known wet process method or a dry-type approach can be used. After melting the partial-hydrolysis condensate of the aforementioned tetrapod alkoxysilane in a suitable solvent, or distributing to a dispersion medium as an example of such a surface treatment method, for example and mixing the aforementioned calcium-carbonate powder in this solution or distributed liquid, the technique of heating and drying is mentioned. As an aforementioned solvent or an aforementioned dispersion medium, toluene, a xylene, etc. are mentioned, for example. As conditions of heating and xeransis, it is good at 80-200 degrees C in about 0.5 - 10 hours, for example.

[0027] Moreover, as the technique of another surface treatment, after mixing a part or all (usually 30% or more, especially 50% or more) of the aforementioned (A) component, and the partial-hydrolysis condensate of calcium-carbonate powder and tetrapod alkoxysilane, the method of heat-treating this mixture is mentioned, for example. As conditions of heat-treatment, it is good at 80-200 degrees C in about 0.5 - 10 hours, for example. Thus, in the mixed process or kneading process of each component in the manufacturing process of the constituent of this invention, since the technique of mixing and carrying out surface treatment of the partial-hydrolysis condensate of a part or all of (A) component, calcium-carbonate powder, and tetrapod alkoxysilane can perform surface treatment of calcium-carbonate powder simultaneously, it is desirable at the point that rationalization by energy saving, process time compaction, etc. can be attained.

[0028] Although the amount of the partial-hydrolysis condensate used of the aforementioned tetrapod alkoxysilane is doubled with the specific surface area of calcium-carbonate powder, or other characters and can be adjusted suitably, it is usually good per calcium-carbonate powder 100 weight section and at 0.1 - 20 weight section grade. It prevents that form a siloxane coat in the front face of the aforementioned calcium-carbonate powder, and an alkali component is

eluted from calcium-carbonate powder with such surface treatment.

[0029] the amount of the calcium-carbonate powder used which carried out surface treatment -- per alkenyl machine inclusion diorganopolysiloxane 100 weight section of a component (A) -- usually -- 5 - 200 weight section -- it is 10 - 100 weight section preferably The thermal resistance of the hardened material obtained if there is too little this amount used, and an electric insulation may become inadequate, the viscosity of the hardenability silicone rubber constituent obtained if many [conversely / too] may rise, and a fluidity may become bad.

[0030] In the constituent of other component this inventions, they are the aforementioned component (A) and a component (B). In addition to a component (C) and a component (D), un-reinforcing nature inorganic bulking agents, such as reinforcement nature inorganic bulking agent; silicic-acid calcium, such as a fumed silica and fumed titanium dioxide, a titanium dioxide, ferric oxide, and carbon black, etc. can be added if needed. The amount of these inorganic bulking agents used is usually 0 - 200 weight section per total quantity 100 weight section of the component except this inorganic bulking agent.

[0031] A hardenability silicone rubber constituent and the constituent of the hardened material this invention are a component (A), (C), a component (D), and a component (A) like a usual hardenability silicone rubber constituent. And the 2 so-called liquid type constituent which the aforementioned component is divided [constituent] into 2 liquid like a component (B), and mixes and stiffens these 2 liquid at the time of use is sufficient. Moreover, it is a component (A) when using the technique of performing surface treatment of calcium-carbonate powder at the same time it mixes a part or all of (A) component, and the partial-hydrolysis condensate of calcium-carbonate powder and tetrapod alkoxy silane. What is necessary is to mix a part or all, the partial-hydrolysis condensate of alkoxy silane, and calcium-carbonate powder, to prepare beforehand what heat-treated this mixture, and just to consider as the 2 liquid type constituent which makes this 1 liquid. Moreover, the 1 so-called liquid type constituent which mixed each component of component (A) - (D), and added little hardening inhibitors (for example, acetylene alcohol etc.) etc. to this constituent is sufficient. Moreover, an epoxy-group inclusion polysiloxane compound and an ester siloxane compound can be blended as occasion demands in addition to the aforementioned component in order to raise the adhesive property of a constituent. Thus, the constituent of this invention obtained has the good fluidity. And the hardened material of this invention hardens the aforementioned constituent, and is obtained. Although it is the same as that of a well-known addition-reaction hardening type silicone rubber constituent, and is good, for example, being enough hardened also in ordinary temperature as hardening conditions, you may heat if needed. The hardened material of such this invention is excellent in thermal resistance and an electric insulation.

[0032]

[Example] Below an example and the example of a comparison are shown, and this invention is explained to it still concretely.

The example 1 following formula (4): $\text{Vi}(\text{Me})_2 \text{Si}(\text{OSiMe}_2)_n \text{OSi}(\text{Me})_2 \text{Vi}$ (4)

The straight chain-like organopolysiloxane 40 weight section of the vinyl-group inclusion expressed with (Me is the same as that of the above among the

formula, Vi is a vinyl group, and n is the number with which the viscosity in 25 degrees C of this siloxane serves as 1000cSts), The straight chain-like organopolysiloxane 60 weight section of the chain both-ends vinyl-group blockade which is the number with which viscosity [in 25 degrees C in n] serves as 5000cSts in the aforementioned general formula (4), The product made from Japanese *****, FP, 1.2 micrometers of mean particle diameters, the calcium-carbonate [alkali component content:0.02 % of the weight (calcium-hydroxide conversion)] 40 weight section, After kneading the partial-hydrolysis condensate (40 % of the weight of SiO₂ contents) [made in [COL Coat] and ethyl (tradename) poly silicate 40T] 2 weight section of a tetrapod ethoxy silane with 3 rolls, this kneading object was kneaded at 160 degrees C the back for 3 hours using the planetary mixer. Next, the methyl-hydrogen-polysiloxane (content:0.8 mol / 100g of hydrogen atom (SiH base) which is combined with silicon atom) 2 weight section, and the octyl alcohol denaturation solution (2 % of the weight of platinum contents) 0.02 weight section of a chloroplatinic acid were added and stirred in this kneading object, and the hardenability silicone rubber constituent was obtained. as a result of measuring the viscosity of this constituent using B type rotational viscometer, it was 36P (below a poise (25 degrees C) -- the same) About the obtained constituent, the following extraction water electric conductivity, a hydrogen gas yield, the rubber physical properties (hardness, tensile strength, elongation) of a hardened material, the electrical property (a volume resistivity, a dielectric breakdown voltage, a dielectric constant, dielectric dissipation factor) of a hardened material, and the thermal resistance of a hardened material were measured, and the foaming status of a hardened material was observed. A result is shown in Table 1.

The constituent 20 weight section obtained the degree of extraction water electrical conduction, the best toluene 40 weight section, and the pure-water 100 weight section were supplied to the separating funnel, after shaking this for 1 hour, it put, and the aqueous phase was isolated preparatively from this. And the electric conductivity (μS/cm) of the water isolated preparatively was measured using ***** meter.

the amount of hydrogen gas which sealed 5g of hydrogen gas yield profit ****s constituents in the 500ml head-space bottle, and left at the room temperature for 48 hours, and was generated in this bottle was measured by the gas chromatograph

after having slushed the rubber physical-properties profit ****s constituent of a hardened material into 150mmx100mmx2mm metal mold and carrying out the vacuum degassing of this, it heated at 150 degrees C for 4 hours, and the sheet-like hardened material was obtained About the obtained hardened material, it is JIS. K It measured according to 6301. In addition, hardness used A type of a spring formula hardness tester.

after having slushed the electrical property profit ****s constituent of a hardened material into 150mmx100mmx1mm metal mold and carrying out the vacuum degassing of this, it heated at 150 degrees C for 4 hours, and the sheet-like hardened material was obtained About the obtained hardened material, it is JIS. C It measured according to 2123.

It is JISK about hardness after heating the hardened material of the shape of same sheet as what was used for measurement of the rubber physical properties of the heat-resistant above of a hardened material at 250 degrees C

for 24 hours, and hardness after heating at this temperature for 48 hours. It measured according to 6301 (A type of a spring formula hardness tester is used).

the foaming status profit ****s hardened material of a hardened material was observed visually, and the existence of a foaming was judged

In example 2 example 1, it replaced with the partial-hydrolysis condensate of a tetrapod ethoxy silane, and the hardenability silicone rubber constituent was obtained like the example 1 except having used the partial-hydrolysis condensate [weight-average-molecular-weight (Mw):788] 2 weight section of a tetramethoxy silane. It was 37P as a result of measuring the viscosity of this constituent using B type rotational viscometer. And an extraction water electric conductivity, a hydrogen gas yield, the rubber physical properties (hardness, tensile strength, elongation) of a hardened material, the electrical property (a volume resistivity, a dielectric breakdown voltage, a dielectric constant, dielectric dissipation factor) of a hardened material, and the thermal resistance of a hardened material were measured like the example 1 using the obtained constituent, and the foaming status of a hardened material was observed. A result is shown in Table 1.

In example of comparison 1 example 1, it replaced with the partial-hydrolysis condensate of a tetrapod ethoxy silane, and the hardenability silicone rubber constituent was obtained like the example 1 except having used the hexamethyldisilazane 3 weight section. It was 41P as a result of measuring the viscosity of this constituent using B type rotational viscometer. And an extraction water electric conductivity, a hydrogen gas yield, the rubber physical properties (hardness, tensile strength, elongation) of a hardened material, the electrical property (a volume resistivity, a dielectric breakdown voltage, a dielectric constant, dielectric dissipation factor) of a hardened material, and the thermal resistance of a hardened material were measured like the example 1 using the obtained constituent, and the foaming status of a hardened material was observed. A result is shown in Table 1.

In example of comparison 2 example 1, the hardenability silicone rubber constituent was obtained like the example 1 except having not used the partial-hydrolysis condensate of a tetrapod ethoxy silane. It was 46P as a result of measuring the viscosity of this constituent using B type rotational viscometer. And an extraction water electric conductivity, a hydrogen gas yield, the rubber physical properties (hardness, tensile strength, elongation) of a hardened material, the electrical property (a volume resistivity, a dielectric breakdown voltage, a dielectric constant, dielectric dissipation factor) of a hardened material, and the thermal resistance of a hardened material were measured like the example 1 using the obtained constituent, and the foaming status of a hardened material was observed. A result is shown in Table 1.

In example of comparison 3 example 1, it replaced with the partial-hydrolysis condensate of a tetrapod ethoxy silane, and the hardenability silicone rubber constituent was obtained like the example 1 except having used the monomethyl triethoxysilane 3 weight section. It was 42P as a result of measuring the viscosity of this constituent using B type rotational viscometer. And an extraction water electric conductivity, a hydrogen gas yield, the rubber physical properties (hardness, tensile strength, elongation) of a hardened material, the electrical property (a volume resistivity, a dielectric breakdown voltage, a dielectric constant, dielectric dissipation factor) of a hardened

material, and the thermal resistance of a hardened material were measured like the example 1 using the obtained constituent, and the foaming status of a hardened material was observed. A result is shown in Table 1.

[0033]

[Table 1]

	実施例 1	実施例 2	比較例 1	比較例 2	比較例 3
粘度 (P)	36	37	41	46	42
抽出水電導度 ($\mu\text{S}/\text{cm}$)	9	10	50	50	51
水素ガ発生量 ($\mu\text{L}/\text{g}$)	39	42	190	200	190
硬さ	56	57	53	52	53
引張強さ (kgf/cm^2)	68	60	40	42	41
伸び (%)	120	110	100	100	110
体積抵抗率 ($\Omega \cdot \text{cm}$)	2×10^{15}	3×10^{15}	8×10^{14}	9×10^{14}	1×10^{15}
絶縁破壊 (kV/mm)	29	28	26	26	25
誘電率 (50Hz)	3.34	3.29	3.42	3.47	3.49
誘電正接 (50Hz)	8.1×10^{-4}	8.3×10^{-4}	1.3×10^{-3}	8.2×10^{-4}	1.1×10^{-3}
耐熱性	250 °C/ 24hr後の 硬さ	66	67	67	67
	250 °C/ 48hr後の 硬さ	71	72	72	72
発泡の有無	無し	無し	有り	有り	有り

The constituent (the example 1 and two references) of this invention is excellent also in the physical properties of the hardened material which there are few yields of hydrogen gas and is obtained as compared with the thing which used the silazane (example of comparison 1 reference), and the carbon functional silane (example of comparison 3 reference), and the thing (example of comparison 2 reference) which heat-treated the calcium carbonate by the silicone oil as a surface treatment agent of a calcium carbonate as shown in the evaluation table 1.

[0034]

[Effect of the Invention] this invention can offer the calcium-carbonate restoration addition-reaction hardening type silicone rubber constituent with very few yields of the hydrogen gas under store. And this constituent is excellent in the fluidity, and is excellent in electrical properties, such as the thermal resistance after hardening, and an electric insulation, and rubber physical properties. Moreover, the manufacture technique of this invention can attain rationalization by energy saving, process time compaction, etc. in a manufacturing process.

[Translation done.]

JAPANESE

[JP,10-060281,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE
INVENTION TECHNICAL PROBLEM MEANS EXAMPLE

[Translation done.]

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